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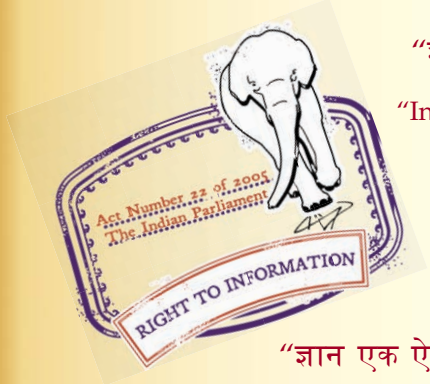
IS 7069 (2001): Benzothiazyl-2-Cyclohexyl Sulphenamide (CBS) [PCD 13: Rubber and Rubber Products]



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"Knowledge is such a treasure which cannot be stolen"

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IS 7069 : 2001

(Reaffirmed - 2012)

भारतीय मानक

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(दूसरा पुनरीक्षण)

Indian Standard

BENZOTHIAZYL-2-CYCLOHEXYL SULPHENAMIDE (CBS) — SPECIFICATION

(Second Revision)

ICS 83.040.01

BUREAU OF INDIAN STANDARDS
MANAK BHAVAN, 9 BAHADUR SHAH ZAFAR MARG
NEW DELHI 110002

December 2001

Price Group 3

FOREWORD

This Indian Standard (Second Revision) was adopted by the Bureau of Indian Standards, after the draft finalized by the Rubber Products Sectional Committee had been approved by the Petroleum, Coal and Related Products Division Council.

Benzothiazyl-2-Cyclohexyl sulphenamide (CBS) is a well known accelerator with a delayed onset of cure for various applications in the rubber industry.

It is soluble in benzene, carbon tetrachloride, acetone, ethanol and insoluble in water. In order to facilitate proper appreciation of this sulphenamide class accelerator in the rubber industry, this standard indicating chemical requirements and technological evaluation procedure has been brought out.

Like all other sulphenamide class accelerators, this sulphenamide accelerator is sensitive to degradation and loss of activity on long storage periods. With time, this material degrades releasing amine and exhibiting a loss in assay and increase in methanol insoluble contents due to formation of MBTS. Use of degraded sulphenamide accelerator in rubber compounds will cause decrease in scorch safety, cure rate and state of cure. Heat, humidity and acidic conditions of storage increase the rate of degradation of sulphenamide accelerators and hence this type of material should be stored in a cool and dry place and away from any acidic material or atmosphere.

This standard was published in 1973 and revised in 1986. First revision was prepared in order to include a more accurate method for determination of assay based on ultraviolet spectroscopy. In this version (second revision) description of the material has been modified. Characteristics and requirement for melting point sulphated as, volatile matter and free amine have been tightened. The characteristics for moisture has been deleted.

The material being a reactive chemical, all persons concerned with handling of this chemical should be properly informed about the nature of the material, possible effect on direct contact, inhalation and accidental ingestion.

The composition of the Committee responsible for formulation of this standard is given in Annex F.

For the purpose of deciding whether a particular requirement of this standard is complied with the final value, observed or calculated, expressing the result of a test or analysis, shall be rounded off in accordance with IS 2 : 1960 'Rules for rounding off numerical values (*revised*)'. The number of significant places retained in the rounded off value should be the same as that of the specified value in the standard.

Indian Standard

BENZOTHIAZYL-2-CYCLOHEXYL SULPHENAMIDE (CBS) — SPECIFICATION

(Second Revision)

1 SCOPE

This Standard prescribes the requirements and method of sampling and test for benzothiazyl-2-cyclohexyl sulphenamide (CBS) intended for use in rubber compounding as a delayed action accelerator for vulcanization of rubber/rubber compounds. This standard is applicable for materials in pellet or powder (uncoated/coated) form.

2 REFERENCES

The following Indian Standards contain provision which through reference in this text, constitute provisions of this standard. At the time of publication, the edition indicated were valid. All standards are subject to revision, and parties to agreements based on the standard are encouraged to investigate the possibility of applying the most recent editions of the standards indicated below:

IS No.	*Title*
1070 : 1992	Reagent grade water (*third revision*)
1675 : 1971	Stearic acid, technical (*first revision*)
3399 : 1993	Zinc oxide for rubber industry (*second revision*)
3400 : (Part 1) : 1987	Methods of test for vulcanized rubbers: Tensile stress — Strain properties (*second revision*)
(Part 2) : 1995	Hardness (*second revision*)
3660 : (Part 7) : 1989	Methods of test for natural rubber: Determination of Money viscosity (NR:8) (*second revision*)
(Part 8) : 2000	Mixing and vulcanizing of rubber in standard compound (NR:9) (*second revision*)
4588 : 1986	Rubber, raw natural (*third revision*)
6918 : 1972	Mercaptobenzothiazole
7086 (Part 1) : 1973	Methods of sampling and test for rubber compounding ingredients
8851 : 1994	Sulphur for rubber industry (*first revision*)

3 REQUIREMENTS

3.1 Description

The material shall be in the form of light cream or tan coloured pellets or granules or powder having relative density of about 1.3 (for guidance only) and shall be free from any visible impurities. The material in the form of pellets or granules or powder may contain dedusting or binding agents approximately 2.0 percent by mass. The manufacturer is required to specify the solvent to be used for extracting the coating material or binder and the material shall be made free from coating material or binder by extracting with the suitable solvent prior to testing for the requirements of methanol insolubles specified in Table 1.

3.2 The material shall also comply with the requirements given in Table 1 when tested according to the procedures given in col 4 of the Table 1.

Table 1 Requirements of Benzothiazyl 1-2-Cyclohexyl Sulphenamide (CBS)

Sl No.	Characteristics	Requirements	Method of Test, Ref to	
			Annex of this Standard	Annex of IS 6918
(1)	(2)	(3)	(4)	(5)
i)	Melting Point, °C			
	a) Appearance of first droplet, *Min*	97	—	C
	b) Completion of melting,	100–107	—	C
ii)	Sulphated ash, percent by mass, *Max*	0.3	—	F
iii)	Copper content, ppm, *Max*	20	—	G
iv)	Volatile matter, percent by mass, *Max*	0.5	A	—
v)	Free amine, percent by mass, *Max*	0.5	B	—
vi)	Assay, percent by mass, *Min*		C	—
	a) For uncoated material	96.0		
	b) For coated / bound material	94.0		
vii)	Methanol insolubles, percent by mass, *Max*	0.5	D	—

NOTE — All tests shall be carried out within 15 days of the receipt of material by the purchaser.

3.3 Compounding

The material when compounded and tested according to the procedure given in Annex E shall have performance characteristics comparable to standard material satisfying the requirements of **3.1** and **3.2**.

4 PACKING AND MARKING

4.1 Packing

The material shall be packed securely so as to avoid exposure to air, moisture and acidic atmosphere. The

material should accompany with Material Data Supply Sheet (MSDS). The exact mode of packing shall be as agreed to between the purchaser and the supplier.

4.2 Marking

Each package shall be marked with the following :

a) Name of the material;
b) Indication of source of manufacture;
c) Net mass of the material;
d) Month and year of the manufacture; and
e) Lot/batch number.

4.2.1 *BIS Certification Marking*

The packages may also be marked with the Standard Mark.

4.2.1.1 The use of the Standard Mark is governed by the provisions of the *Bureau of Indian Standards Act*, 1986 and the Rules and Regulations made thereunder. The details of conditions under which the licence for the use of the Standard Mark may be granted to manufacturers or producers may be obtained from the Bureau of Indian Standards.

5 SAMPLING

5.1 The representative samples of the material shall be drawn as prescribed in IS 7086 (Part 1).

5.2 Number of Tests

5.2.1 Test for copper (*see* Table 1) shall be conducted on individual sample.

5.2.2 Test for all other characteristics shall be conducted on a composite sample.

5.3 Criteria for Conformity

5.3.1 *Copper*

The mean and range of test results for copper shall be calculated as follows :

$$\text{Mean } (\overline{X}) = \frac{\text{Sum of test results}}{\text{Number of test results}}$$

Range (R) = Difference between the maximum and minimum value of the test results

The lot shall be deemed to have satisfied the requirement of the specification if:

$$\overline{X} + 0.620R \leq 20$$

5.3.2 *Composite Samples*

In respect of all other characteristics the lot shall be considered as conforming to the specification if the composite samples satisfies each of these requirements.

6 QUALITY REAGENTS

Unless specified otherwise 'pure chemicals' and distilled water (*see* IS 1070) shall be employed in tests.

NOTE — 'Pure chemicals' shall mean chemicals that do not contain impurities Which affect the results of analysis.

ANNEX A

[*Table* 1, *Sl No.* (iv)]

DETERMINATION OF VOLATILE MATTER

A-1 APPARATUS

Glass petri dish, 12 cm in diameter having 40 ml capacity.

A-2 PROCEDURE

Weigh 25 g accurate to 1 mg of the powdered material and spread it evenly on the dish. The dish is placed in an oven maintained at 65 °C for one hour. The loss in mass is recorded.

A-3 CALCULATION

$$\text{Volatile matter, percent by mass} = \frac{M_1 - M_2}{M_1} \times 100$$

M_1 = mass in g of the sample taken; and
M_2 = mass in g of the sample after heating.

ANNEX B

[*Table* 1, *Sl No.* (v)]

DETERMINATION OF FREE AMINE

B-1 PROCEDURE

Weigh approximately 5 g of the material accurately to 1 mg, and put in a 300-ml Erlenmeyer flask. Add 150 ml of *iso*propanol toluene mixture (5:3) to dissolve the material. The solvent mixture of *iso*propanol-toluene should be neutralized with respect to bromophenol blue to a greenish yellow end point before use. After adding a few drops of bromophenol blue indicator the solution is titrated with 0.1N hydrochloric acid till the appearance of yellow colouration.

B-2 CALCULATION

$$\text{Free amine percent by mass} = \frac{0.99\ V}{M}$$

where

V = volume in ml of 0.1 N hydrochloric acid used, and

M = mass in g of the material taken for the test.

ANNEX C

[*Table* 1, *Sl No.* (vi)]

DETERMINATION OF ASSAY

C-1 SCOPE

This work instruction is applicable for assessing the purity of different sulphenamide type of accelerators (for example CBS) used in rubber and allied industries.

C-2 ABBREVIATION/DEFINITION

Reducing solution — *Iso*propanol/toluene solvent saturated with hydrogen sulphide at 25°C.

C-3 APPARATUS

Burette (50 ml), H_2S trap train, magnetic stirrer, kipp's apparatus for H_2S gas generation, reagent bottle, magnetic stirrer bar, stoppered conical flask (250 ml), glass tubing, two hole stopper, volumetric flask, safety, glasses, laboratory fume cupboard.

C-4 MATERIAL

Bromophenol blue indicator solution, sodium hydroxide, tris buffer, hydrochloric acid (0.1N), primary titrant (0.25 N to 0.35 N), *iso*propanol, toluene, ferrous sulphide, hydrochloric acid (2N), reducing solution, 12 percent NaOH solution, 25 percent NaOH solution, distilled water.

C-5 METHOD

Hydrogen sulphide is a toxic gas and should only be handled in a fume cupboard. The H_2S trap train should be used for catching unused H_2S when preparing the reagent. Toluene and *iso*propanol are highly inflammable, should be handled with appropriate precaution.

C-5.1 Preparation of Reducing Solution

C-5.1.1 Set Kipp's apparatus for H_2S generation in a fume cupboard. Use a 1 000-ml reagent bottle for saturating *iso*propanol/toluene solvent and add a magnetic stir bar. Take a two hole plastic stopper and insert the outlet tube of H_2S into one hole and another plastic tube into the other hole, so that the outlet tube of H_2S should be below will be above the level of *iso*propanol/toluene. Set the reagent bottle onto a magnetic stirrer. Insert the outlet tube of the reagent bottle, through a two hole rubber stopper, into an empty 500-ml conical flask. Insert another plastic tube into the same flask through another hole. Insert the outlet tube of the conical flask into a bottle contain 12 percent NaOH solution through a two hole plastic stopper. Inlet tube should be below the level of the NaOH solution. Insert another plastic tube into the same bottle through another hole in such a way so that the plastic tube will be above the level of 12 percent NaOH solution.

C-5.1.2 Insert plastic tubing in 25 percent NaOH solution containing bottle and distilled water bottle respectively in the same way as 12 percent NaOH solution containing bottle. This type of connection forms H_2S trap train.

C-5.1.3 Add ferrous sulphide and dilute hydrochloric acid (2N) into the Kipp's apparatus. As soon as ferrous sulphide will come in contact with hydrochloric acid H_2S gas generation will take place.

C-5.1.4 Sparge H_2S gas into *iso*propanol/toluene solvent. Stir the *iso*propanol/toluene solvent continuously until saturation is achieved. Saturation will be indicated by an increase in bubble rate of H_2S gas in the *iso*propanol/toluene solvent as well as yellow colour in the 12 percent NaOH solution. Store the reducing solution loosely capped at ambient temperature.

C-5.2 Standardization of Primary Titrant

C-5.2.1 Take 1.3 - 1.5 gm TRIS buffer in a 250 ml conical flask. Add 10 ml distilled water into the conical flask and place on magnetic stirrer until the TRIS buffer get dissolved.

C-5.2.2 Add 150 ml *iso*propanol/toluene solvent into the conical flask. Add 5 drops of bromophenol blue and titrate with the primarily titrant to the first yellow end point. Calculate the normality of the primary titrant as follows:

$$N = \frac{T}{(V_{HCl})(0.121\ 14)}$$

where

T = mass of TRIS buffer,

V_{HCl} = volume of hydrochloric acid required for titration, and

0.121 14 = equivalent mass of TRIS buffer – 1000.

C-6 PROCEDURE

Take 2.0 gm homogenized accelerator sample into a 250 ml stoppered conical flask add magnetic stirrer bar. Add 50 ml *iso*propanol/toluene solvent, stopper and

dissolve by slowly stirring on magnetic stirrer (Take care not to splash solids onto the sides of flask). Allow at least 10 min stirring for complete dissolution of accelerator. Add 5 drops of bromophenol blue indicator solution and titrate the excess base with 0.1 N HCl to the first yellow end point. Immediately after neutralizing the excess base, add 125 ml of reducing solution, replace the stopper and set aside for minimum of 90 min at ambient temperature to complete sulphenamide reduction. Complete reduction can be identified as wine or rust colour of the sample solution. While stirring rapidly with the magnetic stirrer titrate the liberated amine with the primary titrant to the first yellow end point. Two drops of additional indicator added near the end point will intensify the colour change. Record the volume of the primary titrant as V_a and use in the calculation. Determine the percent assay as follows:

$$A = \frac{V_a \times V_{HCl} \times MS}{S} \times 100$$

where

V_a = volume of primary titrant
V_{HCl} = normality of primary titrant
MS = for CBS : 0.2644.
S = sample mass

ANNEX D

[*Table* 1, *Sl No* (vii)]

DETERMINATION OF METHANOL INSOLUBLES

D-1 PROCEDURE

D-1.1 Weigh 4 g of the material to the nearest milligram and put in a 400 ml beaker. Add 250 ml of methanol into it. Stir it at a room temperature for 30 minutes with the help of a magnetic stirrer. Allow the residue to settle. Decant the bulk of the supernatent liquid. Transfer the residue as thick slurry on to a previously weighed centred glass crucible (porosity No. 3). Wash the cake two or three times with 25 ml of methanol and then suck it dry. Dry the crucible at 100°C and weigh.

D-1.2 In case of granular / pellet material, the material shall be powdered before testing.

D-2 CALCULATION

Methanol Insolubles, percent by mass = mass of the residue x 25

ANNEX E

(*Clause* 3.3)

METHODS FOR COMPOUNDING AND TESTING OF BENZOTHIAZYL-2-CYCLOHEXYL SULPHENAMIDE (CBS)

E-1 TEST COMPOUND

As a guidance, the following test compound may be used for testing formance properties of benzothiazyl-2-cyciohexyl sulphenamide (CBS) in rubber compound:

	Parts by Mass
Natural rubber, Grade A (conforming to IS 4588)	100
Carbon black, HAF	45
Zinc oxide (conforming to IS 3399)	5
Mineral oil (aromatic) plasticizer	5
Stearic acid (conforming to IS 1675)	3
Sulphur (conforming to IS 8851)	2.5
Benzothiazyl-2-cyclohexyl sulphenamide (CBS)	0.5

E-2 COMPOUNDING

Follow the procedure prescribed in IS 3660 (Part 8).

E-3 TESTS

E-3.1 The tests given below are recommended for each test sample. The approved sample shall also be tested side by side using the same masterbatch, that is, compound excluding accelerator only.

E-3.1.1 Mooney scorch test shall be done at 120°C in accordance with the method prescribed in IS 3660 (Part 7).

E-3.1.2 Tensile strength, modulus at 300 percent elongation, elongation at break at different cures at 141°C (from below to above the expected optimum cure) shall be tested in accordance with the method described in IS 3400 (Part 1).

E-3.1.3 Hardness on optimum cure at 141°C shall be tested in accordance with the method prescribed in IS 3400 (Part 2).

E-4 RESULT

The values obtained with the test sample shall not vary by more than 20 percent for Mooney scorch and ± 10 percent for all the other characteristics from those obtained with the approved sample.

ANNEX F

(Foreword)

COMMITTEE COMPOSITION

Rubber Products Sectional Committee, PCD 13

Organization	*Representative(s)*
Apar Industries Ltd, Mumbai	SHRI MANU M. PATEL (***Chairman***)
All India Rubber Industries Association, Mumbai	SHRI M. M. PATEL SHRI K. D. SHAH (*Alternate*)
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(Continued on page 6)

(Continued from page 5)

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Review of Indian Standards

Amendments are issued to standards as the need arises on the basis of comments. Standards are also reviewed periodically; a standard along with amendments is reaffirmed when such review indicates that no changes are needed; if the review indicates that changes are needed, it is taken up for revision. Users of Indian Standards should ascertain that they are in possession of the latest amendments or edition by referring to the latest issue of 'BIS Handbook' and 'Standards: Monthly Additions'.

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Amendments Issued Since Publication

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